



Europäisches
Patentamt

European
Patent Office

Office européen
des brevets

REC'D 04 NOV 2003

WIPO

PCT

Bescheinigung

Certificate

Attestation

Die angehefteten Unterla-
gen stimmen mit der
ursprünglich eingereichten
Fassung der auf dem näch-
sten Blatt bezeichneten
europäischen Patentanmel-
dung überein.

The attached documents
are exact copies of the
European patent application
described on the following
page, as originally filed.

Les documents fixés à
cette attestation sont
conformes à la version
initialement déposée de
la demande de brevet
européen spécifiée à la
page suivante.

Patentanmeldung Nr. Patent application No. Demande de brevet n°

02078734.7

BEST AVAILABLE COPY

CERTIFIED COPY OF
PRIORITY DOCUMENT

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
p.o.

**PRIORITY
DOCUMENT**
SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

R C van Dijk



Anmeldung Nr:
Application no.: 02078734.7
Demande no:

Anmeldetag:
Date of filing: 04.09.02
Date de dépôt:

Anmelder/Applicant(s)/Demandeur(s):

NV. Topchim SA.
Nijverheidsstraat 92
2160 Wommelgem
BELGIQUE

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se référer à la description.)

Vapor screen composition

In Anspruch genommene Priorität(en) / Priority(ies) claimed / Priorité(s)
revendiquée(s)
Staat/Tag/Aktenzeichen/State/Date/File no./Pays/Date/Numéro de dépôt:

Internationale Patentklassifikation/International Patent Classification/
Classification internationale des brevets:

D21H/

Am Anmeldetag benannte Vertragsstaaten/Contracting states designated at date of
filing/Etats contractants désignées lors du dépôt:

AT BE BG CH CY CZ DE DK EE ES FI FR GB GR IE IT LI LU MC NL PT SE SK TR

0001EP/P0/

-1-

VAPOR SCREEN COMPOSITION5 Field of the invention

The present invention relates to a water-dispersible composition, with a low permeability for gas and moisture and a moisture-proof paper made using the foregoing composition.

10 Background of the invention

Recently, from the standpoint of saving resources, a demand of collecting used papers and regenerating papers again after dissolving the papers in water has been increased. It is required for the papers meeting the demand to have a so-called easily macerating property, that is, when the paper is dissolved in water the paper is easily macerated into fine fibrous forms.

15 A composition for making an easily macerating moisture proof paper is known from US 5,527,623. Example 8 of US 5,527,623 describes aqueous emulsion comprising components (A), (B) and (C), wherein component (A) is an ethylene-vinyl chloride series copolymer comprising 20% by weight ethylene, 41% by weight vinyl chloride, and 39% by weight vinyl acetate having a glass transition temperature of from 0° C, component (B) is an emulsion of a rosin ester having a softening point of 125° C and component (C) is an emulsion of a wax having a melting point of 57° C. Applies on a paper having a base weight of 80 g/m² in an amount of 20g/m² the coated paper had a moisture permeability of 19 g/m²-24 hr.

25

Summary of the invention

The object of the present invention is to provide a composition, for making a water repellent paper with a low permeability for moisture.

30 The water-dispersible composition of the present invention comprises components (A) to (H), in the following weight (wt) percentages;

- (A) 5 to 89 wt % of polyvinylbutyral (PVB),
- (B) 1 to 6 wt % of a styrene maleic anhydride polymer (SMA),
- (C) 10 to 50 wt % of a ethylene acrylate,
- 35 • (D) 0 to 10 wt % of a polyvinyl alcohol or starch

- 2 -

- (E) 0 to 7 wt % of a wax.
- (F) 0 to 20 wt % of Styrene Maleic Imide (SMI)
- (G) 0 to 70 wt % of a Styrene Butadiene Rubber (SBR) latex
- (H) 0 to 30 wt % of talcum

5

Detailed description of the invention

The present invention is described in detail below. The aqueous emulsion of the present invention contains the components (A), (B) and (C) described above as the essential components.

10

Component (A) is 5 to 89 % by weight of PVB in water. If the content of the PVB in the composition of the present invention is below 5 wt %, the paper obtained with such emulsion has poor water repulsive properties and a too low moisture permeability. A composition with more than 89 wt % of PVB has a too high shear resistance for being applied in a high speed paper coating process.

15

A preferred composition comprises 40 to 70 wt % PVB. Paper treated with a composition according to the invention comprising less than 70 wt % of PVB can be macerated with an home mixer using a low mixing speed while a paper casted from a thus obtained pulp exhibits no sticky and agglomerated particles. Paper treated with a composition comprising at least 40 wt % of the PVB turned out to have an excellent

20

resistance against penetration of solvents.

PVB generally is used as an emulsion in water. A suitable concentration of the PVB emulsion is about 50 wt %. A concentration above 50 wt % is hard to process due to its fast drying character.

25

The emulsion comprises PVB particles with a preferred average particle size of between 90 and 1000 nm. In order to stabilize the PVB emulsion, the emulsion further comprise an emulsifier. A suitable emulsifier for PVB in water is e.g. Castor oil or the like.

In general the PVB emulsion comprises at least 20, but more preferably 40 wt % of an emulsifier on 100 wt % of the PVB.

30

Component (B) is a styrene maleic anhydride copolymer (SMA) comprising preferably between 20 and 50 % by weight of maleic anhydride units.

A suitable molecular weight of the SMA is between 80.000 and 150.000 g/mol. The

amount of SMA in the composition of the invention is between 1 and 6 wt %. An amount of at least 1 wt % of SMA improves the rheological behavior of the composition of the present invention. An additional advantage of at least 1 wt % of SMA in compositions comprising up to 7 wt % of wax is that it prevents the migration of a, in particular low melting, wax to the surface of a paper coated with such composition. Compositions with more than 6 wt % of SMA should be avoided because of their hydrophilic character.

Component (C) is an ethylene acrylate copolymer comprising from 5 to 30% by weight of ethylene and a particle size of between 100 and 1000 nm. Preferably the particle size is between 300 and 700 nm. Most preferably the particle size is between 450 and 550 nm. The amount of ethylene acrylate in the composition of the invention is between 10 and 50 wt %. An amount of at least 10 wt % of ethylene acrylate to obtain a paper which is easily sealable with a water based glue, a hot melt or which is thermosealable. Compositions with more than 50 wt % exhibits a too high vapor transmission.

The composition of the invention may further comprise the following components:

- (D) 0 to 10 wt % of a polyvinyl alcohol or starch
- (E) 0 to 7 wt % of a wax.
- 20 • (F) 0 to 20 wt % of Styrene Maleic Imide (SMI)
- (G) 0 to 70 wt % of a Styrene Butadiene Rubber (SBR) latex
- (H) 0 to 30 wt % of talcum

Component (D) comprises up to 10 wt % of polyvinyl alcohol or starch and is generally added to improve the rheological properties of the composition. A suitable polyvinyl alcohol has a relatively high molecular weight, preferably more than 150.000 g/mol and has an as low as possible degree of soapification.

Component (E) is the emulsion of a wax having a melting point of from 50° C. to 100° C., preferably from 50° C. to 70° C. If the melting point thereof is too low, the paper obtained using the aqueous emulsion has poor adhesion properties, while if the melting point thereof is too high, the paper obtained has poor hydrophobic properties. The emulsion of wax which can be used in the present invention is, for example, a commercially available paraffin wax emulsion or microwax emulsion, and if

desired and necessary, a mixture of these emulsions can be used.

Component (F) comprises at most 20 wt % of a styrene maleic imide copolymer (SMI). More than 20 wt % should be avoided because of a too low thermosealability of a paper coated with the composition according to the invention. A preferred SMI copolymer comprises between 20 and 30 wt % maleic monomer units.

Component (H) is a styrene Butadiene Rubber (SBR), which may be added to the composition of the invention in an amount of up to 70 wt %. Above 70 wt % the vapor transmission rate is too high, while the water repulsion becomes too low.

Component (H) is a standard talcum with a preferred particle size of between 15 and 60 micron. Using talcum in the composition of the invention even further decreases the moisture transmission rate.

The aqueous emulsion of the present invention can be produced by, for example, the following method. The PVB emulsion as the component (A) is placed in a vessel equipped with a stirrer and stirred to an extent of not forming bubbles. An aqueous solution of SMA and the emulsion of ethylene acrylate are successively added to the copolymer possibly followed by the components (D) to (H) and followed by stirring for mixing. The aqueous emulsion of the present invention can be thus obtained.

The invention further relates to an easily macerating moisture-proof paper comprising a base paper being coated on at least one surface thereof a layer of an aqueous emulsion comprising a water-dispersible composition according to the invention, wherein the solid content in the aqueous emulsion is from 30 to 65 % by weight. Preferably the solid content is from 45 to 50 % by weight. Within the range from 30 to 60 wt % the viscosity of the composition according to the invention is suitable for processing on a paper coating machine. The viscosity of a composition with 45 to 50 wt % of solid content allows processing on high speed paper coating machines.

The easily macerating moisture-proof paper of the present invention is obtained by coating the aqueous emulsion on at least one surface of a base paper in an amount of from 8 to 25 g/m². If the amount of the aqueous emulsion used is below 8 g/m², the paper obtained has poor moisture-proof property and water repellency by the formation of pinholes and the occurrence of fluffing of the fibers of the base paper, while if the amount of the aqueous emulsion is above 25 g/m² no relevant improvement of the vapor transmission rate is obtained.

A kraft paper, a wood free paper, a corrugated board base paper, etc., can be used as the base paper, but the base paper used in the present invention is not limited to those.

5 For obtaining the easily macerating moisture-proof paper of the present invention, for example, the definite amount of the aqueous emulsion is coated on a base paper with e.g. a blade, a rollercoater or an Airknivecoater, and dried at a temperature of higher than about 125 °C and up to 200 °C. In addition, when the drying temperature is too low, it sometimes happens that the easily macerating moisture-proof paper obtained is inferior in the moisture-proof property.

10 The easily macerating moisture-proof paper of the present invention is most suitably used in a wide field, for example, as a wrapping paper, a water-resistant corrugated fiberboard paper, a wrapping paper for copying paper owing to the excellent characteristics.

15 An extra advantage of the present invention is that the composition according to the invention is free of halogens, has a high resistance against solvents and can be applied on-line on a paper production machine. A paper treated with the composition of the invention exhibits a high a high oxygen barrier, which makes it very suitable for the packaging of food.

20 Experimental

The present invention is explained in more detail by the Examples I to

X

25 Aqueous compositions were obtained with amount of the components (A) to (H) as shown in Table 1. For each of the compositions coated paper was made under the following coating condition: Each coated paper was obtained by coating each aqueous emulsion on a kraft paper having a basis weight as given in table 1 with a roller coater at a coating amount as shown in table 1.

30 The coated paper was dried at 140 °C and items of the vapor transmission rate, the water repellency, the friction coefficient, and the grease resistance were evaluated as follows.

The vapor transmission rate was measures a cup method according to DIN 53122.

35 The water repellency was measured according to the Cobb method during 3 respectively 10 minutes with a kraft paper treated with 8 g/m² of a composition according to table 1.

The grease resistance is measured according to the Kitt test according to 3M

The friction coefficient (C.O.F) was measured with a weight of 1 kg on a variable slope. The ratings "low", "medium" and "high" were given.

5 The results obtained are shown in table 2:

-7-

Table 1

Composition - (wt%)	1	2	3	4	5	6	7	8	9	10	11
PVB	35.00	45.00	38.00	38.00	48.00	40.00	38.00	38.00	38.00	38.00	70
SMA	1.00	0.00	1.00	1.00	0.00	1.50	1.00	1.00	1.00	1.00	1
Ethylene-Acrylate	12.00	15.00	15.00	15.00	15.00	10.00	15.00	15.00	15.00	15.00	25
Wax		1.00	7.00	7.00	3.00	3.50	7.00	7.00	7.00	7.00	2
SMI	2.00	4.00	5.00	6.00	2.00	5.00	4.00	4.00	4.00	4.00	2
SBR	35.00	35.00	33.00	38.00	32.00	30.00	35.00	35.00	35.00	35.00	
Talcum	15.00	0.00	0.00	0.00	0.00	10.00	0.00	0.00	0.00	0.00	

Table 2

Exp.	Formula	Basic paper	Staple treatment and weight	Coating weight m ²	Total weight	Cobb 3 min H ₂ O/gm ²	Cobb 10 min H ₂ O/gm ²	Kiln 3M test	C.O.F. low medium High	Vapor transmission 60% RH at +35°C g/m ² after 24 h DIN	Vapor transmission 90% RH at +38°C g/m ² after 24 h DIN
I		Cardboard 2350	Starch 2g	18g	20g	3	8	14	M	53g/m ²	135g/m ²
II		Cardboard 2350	SMI 40% 2g	18g	20g	6	14	11	L	30g/m ²	50g/m ²
III		Paper 90g	Pretop 601-1.5g	17.5g	20g	3	6	13	H	18g/m ²	28g/m ²
IV		Form.3 callandied	Pretop 601-1.5g	17.5	20g	1	4	14	H	11g/m ²	20g/m ²
V		Cardboard 2350	Starch 2g	10g	12g	5	11	13	H	32g/m ²	64g/m ²
VI		Cardboard 2350	X	18g	18g	3	7	9	L	34g/m ²	70g/m ²
VII		Cardboard 265g	Starch+fluorocel.	20g	21.5g	6	17	14	M	70g/m ²	106g/m ²
VIII		Cardboard 265g	Starch+fluorocel.	18g	17g	0	4	14	M	15g/m ²	19g/m ²
IX		Cardboard 265g	Starch+fluorocel.	19	20g	0	5	13	M	36g/m ²	76g/m ²
X		Cardboard 265g	Starch+fluorocel.	10g	20g	0	6	14	H	45g/m ²	89g/m ²

CLAIMS

1. A water-dispersible composition comprises components (A) to (H), in the following weight (wt) percentages:
 - 5 (A) 5 to 89 wt % of polyvinylbutyral (PVB),
 - (B) 1 to 6 wt % of a styrene maleic anhydride polymer (SMA);
 - (C) 10 to 50 wt % of a ethylene acrylate,
 - (D) 0 to 10 wt % of a polyvinyl alcohol or starch
 - (E) 0 to 7 wt % of a wax.
 - 10 (F) 0 to 20 wt % of Styrene Maleic Imide (SMI)
 - (G) 0 to 70 wt % of a Styrene Butadiene Rubber (SBR) latex
 - (H) 0 to 30 wt % of talcum
2. Composition according to claim 1 ,wherein the amount of component (A) is between 40 and 70 wt %
- 15 3. An easily macerating moisture-proof paper comprising a base paper having coated on at least one surface thereof a layer of an aqueous emulsion comprising a water-dispersible composition according to claim 1 or 2, wherein the solid content in the aqueous emulsion is from 30 to 65 % by weight
4. The paper according to claim 3, wherein the. paper is coated on the at least
20 one surface with a composition according to any of claims 1 to 3, in an amount of from 8 to 25 g/m²

ABSTRACT

The invention relates to a water-dispersible composition for making a water repellent paper with a low permeability for moisture. The composition comprises components (A) to (H), in the following weight (wt) percentages; (A) 5 to 89 wt % of polyvinylbutyral (PVB), (B) 1 to 6 wt % of a styrene maleic anhydride polymer (SMA), (C) 10 to 50 wt % of an ethylene acrylate, (D) 0 to 10 wt % of a polyvinyl alcohol or starch; (E) 0 to 7 wt % of a wax; (F) 0 to 20 wt % of Styrene Maleic Imide (SMI); (G) 0 to 70 wt % of a Styrene Butadiene Rubber (SBR) latex; and (H) 0 to 30 wt % of talcum.

a composition, The invention further relates to an easily macerating moisture-proof paper comprising a base paper having coated on at least one surface thereof a layer of an aqueous emulsion comprising a water-dispersible composition, wherein the solid content in the aqueous emulsion is from 30 to 65 % by weight

THIS PAGE BLANK (USPTO)

THIS PAGE BLANK (USPTO)

PCT Application

EP0309993



**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☒ FADED TEXT OR DRAWING
- ☒ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☐ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☒ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.